HEXAVALENT CHROMIUM in Soil and Water

Colorimetric SW-846 Method 7196A *

HEXAVALENT CHROMIUM in Water

Atomic Absorption, Chelation-Extraction EPA Method 218.4

Table 1. Summary of Contract Required Detection Limits, Holding Times, and Preservation for Hexavalent Chromium (Cr⁺⁶)

Analytical Parameter	Contract Required Detection Limit (CRDL)	Technical and Contract Holding Times ^a	Preservation
Hexavalent Chromium (Cr ⁺⁶)in Water Samples	20 µg/L b	Technical: 24 hours from collection; Contract: 12 hours from receipt at laboratory	Cool to 4°C ±2°C e
Cr ⁺⁶ in Soil Samples ^d	2 mg/kg	Contract: 12 hours from receipt at laboratory	Cool to 4°C ±2°C

- * If the colorimetric method is not suitable, EPA Method 218.4 may be used. If EPA Method 218.4 is designated for analysis, the diphenylcarbazide colorimetric procedure discussed in Section 2.3 of EPA Method 218.4 may NOT be used.
- Laboratories bidding on this analysis must be located within a 3 hour drive from the site of sample collection.
- $^{
 m b}$ 20 µg/L CRDL for analysis by SW-846 Method 7196A
- $^{\circ}$ 10 $\mu g/L$ CRDL for analysis by EPA Method 218.4
- Soil Samples: Follow EPA SW-846 Method 3060A (January 1995) for sample digestion.
- Determine initial pH of water samples.

Data Calculations and Reporting Units:

Calculate the sample results from the standard curve. Calculate soil sample results using the equation provided in Section 7.10.1 of Method 3060A. Report water sample results in concentration units of micrograms per liter (µg/L). Report soil sample results in concentration units of milligrams per kilogram (mg/kg). Cr⁺⁶ concentrations that are less than 10 µg/L or 10 mg/kg to 1 significant figure, and Cr⁺⁶ concentrations that are greater than or equal to 10 µg/L or 10 mg/kg to 2 significant figures.

For rounding results, adhere to the following rules:

- a) If the number following those to be retained is less than 5, round down;b) If the number following those to be retained is greater than 5, round up;
- c) If the number following the last digit to be retained is equal to 5, round down if the digit is even, or round up if the digit is odd.

All records of analysis and calculations must be legible and sufficient to recalculate all sample concentrations and QC results. Include an example calculation in the data package.

Table 2. Summary of Calibration Procedures for Hexavalent Chromium

Calibration Element	Frequency	Acceptance Criteria	Corrective Action
Initial Calibration (minimum blank + 5 points for each analyte) (ICAL) a, b	Initially; whenever required, due to failure of ICV or CCV	r \$ 0.995	Terminate analysis Recalibrate and verify before sample analysis
Initial Calibration Verification (ICV) at midpoint of ICAL (Separate source from ICAL standards)	Daily, prior to sample analysis; immediately following ICAL	±10% from expected concentration	Identify and document problem Reprep ICV and reanalyze all associated samples Recalibrate and reanalyze reprepped ICV and all associated samples
Continuing Calibration Verification (CCV)	Following ICV and before sample analysis; after every 10 samples and end of run	± 10% from expected concentration	Recalibrate and verify Reanalyze samples back to last good CCV
Calibration Blank Verification (ICB, CCB)	After ICV and every CCV	< CRDL	1. Terminate analysis 2. Identify and document the problem 3. Recalibrate, verify and reanalyze all associated samples with results less than 10 times the level of contamination in the blank.
CRDL Verification Standard	After initial CCV/CCB	± 20% from expected concentration	1. Reprep and reanalyze standard 2. Recalibrate and verify

The ICAL standards for SW-846 Method 7196A must include standard concentrations at the CRDL (20 $\mu g/L$) through 500 $\mu g/L$.

The ICAL standards for EPA Method 218.4 must include standard concentrations at the CRDL (10 $\mu g/L$) through 250 $\mu g/L$.

A calibration curve must be prepared with each set of samples.

Table 3. Summary of Internal Quality Control Procedures for Hexavalent Chromium

QC Element	Frequency	Acceptance Criteria	Corrective Action
Laboratory Method Blank (MB)	One per Batch or SDG ^a (1 per 20 samples minimum)	< CRDL	1. If lowest sample concentration is more than 10X the blank conc., no action 2. If samples are non-detected, no action 3. If detected sample concentrations are less than 10X blank conc., all associated samples must be prepared again with another method blank and reanalyzed
Duplicate Sample (DUP)	One per batch or SDG (1 per 20 samples minimum)	RPD <20% for samples >5X CRDL; ± CRDL for samples <5X CRDL	1. Flag associated data with an "*"
Matrix Spike (MS)	One per batch or SDG (1 per 20 samples minimum)	± 25% from expected value	1. Flag associated data with an "N"
Laboratory Control Sample (LCS)	One per batch or SDG (1 per 20 samples minimum)	± 20% from expected concentration	1. Terminate analysis 2. Identify and document the problem 3. Reanalyze all associated samples

^a SDG - Sample Delivery Group - each case of field samples received; or each 20 field samples within a case; or each 14 calendar day period during which field samples in a case are received.

Dilute and reanalyze samples with concentrations exceeding the range of the calibration curve. Results for such reanalyses should fall within the mid-range of the calibration curve. Report results and submit documentation for both analyses.